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IS 3753 (1984): Methods of sampling for alcoholic drinks
[FAD 14: Drinks and Carbonated Beverages]



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IS : 3753 - 1984

Indian Standard
METHODS OF SAMPLING FOR
ALCOHOLIC DRINKS
(*First Revision*)

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

METHODS OF SAMPLING FOR ALCOHOLIC DRINKS

(*First Revision*)

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(*Continued on page 2*)

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Indian Standard

METHODS OF SAMPLING FOR ALCOHOLIC DRINKS

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 20 March 1984, after the draft finalized by the Sampling Methods for Food Products and Agricultural Inputs Sectional Committee had been approved by the Agricultural and Food Products Division Council.

0.2 The alcoholic industry is at present a well organized and established industry in India. Substantial quantities of the products are being exported also. It is, therefore, imperative that there is strict quality control and due consideration is given to sampling procedures which will help in proper and objective evaluation of the various characteristics. This standard lays down the methods of sampling for alcoholic drinks. While evolving this standard, due weightage has been given to the trade practices.

0.3 The sampling procedures given in this standard include, besides lot inspection plan, recommended provisions for process control. Proper quality control during process would substantially reduce quality fluctuations and help the manufacturer in achieving in-built quality of the product. Lot inspection plan would enable the manufacturer or purchaser or other independent inspection and testing agencies to decide the conformity or otherwise of the material to the relevant specification.

0.4 This standard was originally issued in 1967. In this revised version, recommended provisions of process control for the guidance of the manufacturer have been elaborated, the definition of lot and scale of sampling have been suitably modified in the light of experience gained and modified tolerances for net volume given in individual material specifications. The provisions regarding attribute type of requirements and net volume have been appropriately incorporated in the criteria for conformity. A modified design of the weighted sampling can has also been included.

0.5 This standard is intended to introduce uniform methods of sampling for alcoholic drinks. It does not deal with the specifications of the materials, but prescribes only the sampling procedures for determining the conformity of the manufactured products to the specified quality, besides recommended process control provisions, and thus forms a necessary adjunct to the series of Indian Standard specifications for individual alcoholic drinks.

0.6 This standard is subject to the restrictions imposed under the *Prevention of Food Adulteration Act, 1954*, and the rules framed thereunder, and the State Excise Duty Rules which permit the withdrawal of duty free samples for testing. It is recommended that samples for testing by ISI or any other government recognized inspection and testing agency, whenever called for, may also be exempted from excise duty.

1. SCOPE

1.1 This standard prescribes the methods of sampling and criteria for ascertaining the conformity of the alcoholic drinks to the relevant material specifications. It also includes the recommended provisions for process control for the guidance of manufacturers.

2. GENERAL REQUIREMENTS OF SAMPLING

2.1 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed as far as possible:

- a) Samples shall be taken in a protected place not exposed to damp air, dust or soot;
- b) The sampling instrument shall be clean and dry;
- c) To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means;
- d) Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from extraneous contamination;
- e) Samples shall be placed in suitable clean, dry and airtight glass containers.
- f) The sample containers shall be of such a size that sufficient head space to allow for expansion of the liquid, is left after pouring in the sample;
- g) Each sample container shall be sealed airtight with a suitable stopper after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material; and
- h) Samples shall be stored in a cool, dark and dry place.

3. SAMPLING INSTRUMENTS

3.0 The following types of sampling instruments may be used :

- a) Sampling can or weighted sampling can for taking samples from various depths of large tanks, and
- b) Sampling tube.

3.1 Sampling Can — It consists of a metal container of suitable capacity (about 1 litre) attached to a suitable lead. The can has a removable stopper, to which is attached a light chain (*see* Fig. 1). For taking a sample, it is lowered in the tank to the required depth, and the stopper is removed by means of the chain for filling the container.

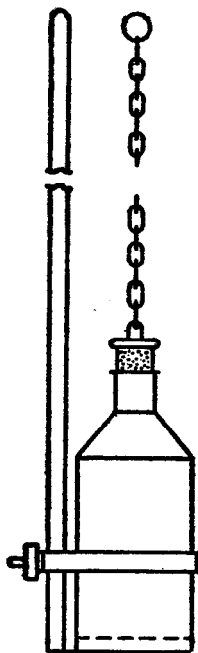


FIG. 1 SAMPLING CAN

3.2 Weighted Sampling Can — This can, shown in Fig. 2, may be made of suitable metal. It shall be of suitable capacity, for example, 1 litre, and of such dimensions that it will pass freely through the tank dip-hatch. The can shall be of such a mass as to sink rapidly in the material to be sampled. The metal used to weigh the apparatus shall either be fitted externally or

be contained in the space provided by fitting the can with a false bottom. The apparatus shall be provided with means to permit filling at any desired level and closure, and shall have a suitable long chain or cord of non-sparking material attached to it.

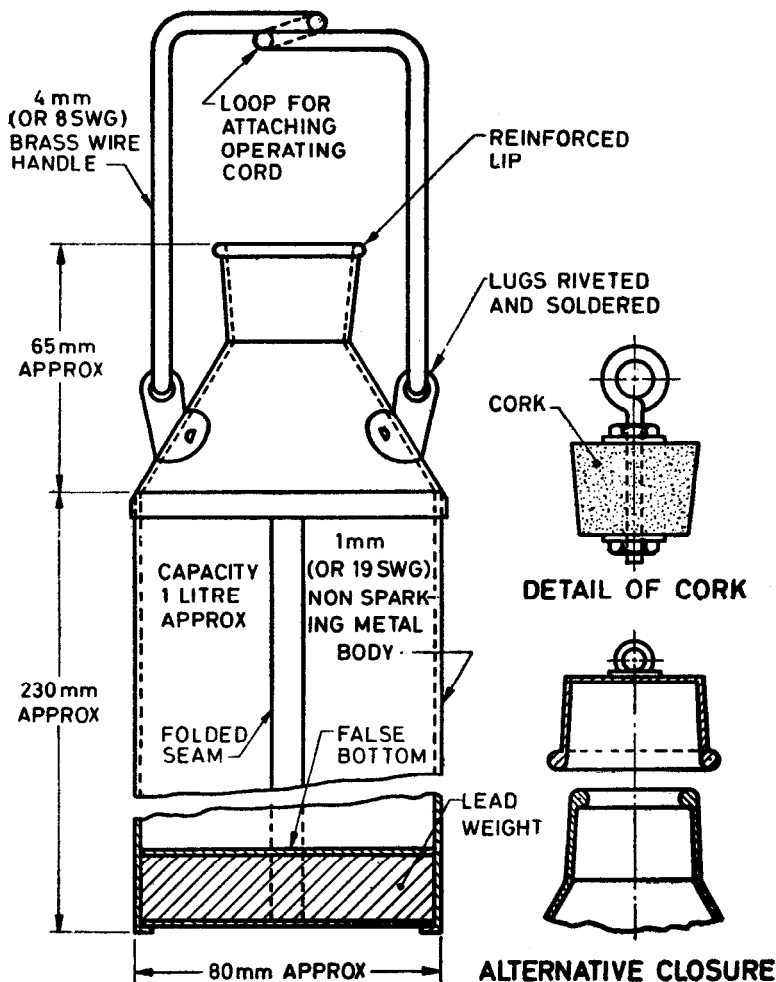


FIG. 2 WEIGHTED SAMPLING CAN

3.3 Sampling Tube — It is made of metal or thick glass and is 20 to 40 mm in diameter and 0.5 to 1.0 m in length (see Fig. 3). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For taking a sample, the tube is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

3.3.1 For small containers, the size of the sampling tube may be altered suitably.

3.4 Materials — All the materials used for fabricating the sampling instruments shall be such as not to contaminate or chemically affect the sample or the material being sampled.

4. PROCESS CONTROL

4.1 One sample from each consignment of raw materials received in the factory shall be tested for important requirements of that material. Adequate

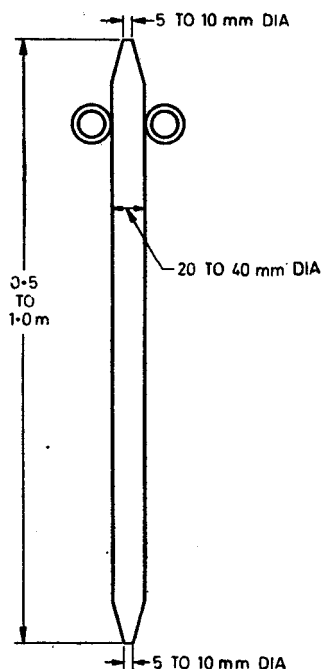


FIG. 3 SAMPLING TUBE

control shall be exercised on the quality of water used for the manufacture and for this purpose IS: 4700-1968* may be referred.

4.2 The manufacturer shall make appropriate checks and take representative samples at intermediate stages of processing so as to control the quality fluctuations and hence ensure that the finished material is of desired quality.

4.3 The inspection levels given below are recommended for routine control over the manufacturing process.

4.3.1 *Fermented Alcoholic Beverages (Beer and Wines)*

4.3.1.1 Beer — The entire quantity of beer manufactured and stored at a time in each bottling vat just prior to bottling shall constitute a batch.

The recommended frequency of testing for various characteristics at different stages of manufacture is given in Appendix A.

4.3.1.2 Wines — The provisions similar to that of beer given in **4.3.1.1** shall apply with the modification that the stage 'wort boiling' shall be deleted and the stage 'Meshing' shall be replaced by 'juice extraction'.

4.3.2 *Distilled Alcoholic Beverages*

4.3.2.1 Whisky, rum, gin and brandy — The quantity of material manufactured at a time in the compounding/dilution/distillation tank shall constitute a batch.

The recommended frequency of testing for various characteristics at different stages of manufacture is given in Appendix B.

5. LOT INSPECTION

5.1 If the manufacturer has maintained an adequate and satisfactory system of quality control in the manufacture of alcoholic drinks, the resulting data and information may be made available to the purchaser alongwith the material supplied to enable him to judge the acceptability of the consignment. When it is not possible to provide this information or if the purchaser so desires, the procedure laid down in the following clauses shall be followed for determining the conformity of material to the requirements of the specification.

5.2 Scale of Sampling

5.2.1 Lot — The quantity of packed alcoholic drink of the same type, belonging to the same batch of manufacture (*see 4.3.1.1 and 4.3.2.1*) and packed in a day, shall constitute a lot.

*Quality tolerances for water for fermentation industry.

5.2.2 For ascertaining the conformity of the material to the requirements of the relevant specification, samples shall be tested from each lot separately.

5.2.3 The number of bottles to be selected from a lot shall depend on the size of the lot and shall be according to Table 1. The bottles selected for net volume according to col 3 of Table 1 shall be in addition those selected according to col 2 of Table 1.

TABLE 1 SCALE OF SAMPLING

No. of BOTTLES IN THE LOT	SAMPLE SIZE	
	Requirements Other Then Net Volume	Net Volume
(1)	(2)	(3)
Up to 5 000	9	36
5 001 „ 10 000	12	36
10 001 „ 15 000	15	72
15 001 and above	21	108

5.2.3.1 These bottles shall be chosen at random from the lot. In order to ensure the randomness of selection, procedures given in IS : 4905-1968* may be followed.

5.2.4 Initially the number of cartons equal to the number of bottles to be taken from the lot in one set (*see 5.3.3*), shall be chosen at random. These cartons thus selected shall be opened and the bottles in these cartons examined visually for the condition of packing, the external appearance and the fill. The lot shall be considered satisfactory for inspection of other characteristics given in the specification, if all the bottles in the cartons opened are found meeting the requirements for these characteristics.

5.2.5 In case any defective bottle is found according to **5.2.4**, twice the number of cartons shall be opened and the bottles examined for similar characteristics. If no defective bottle is found, the lot shall be considered satisfactory for inspection of other characteristics given in the specification.

5.3 Preparation of Test Samples

5.3.1 From each of the cartons opened according to **5.2.4**, three bottles shall be taken from its different layers so as to obtain the required number of bottles in the sample (*see col 2 and 3 of Table 1*).

*Methods for random sampling.

5.3.2 In case the number of cartons to be opened is according to 5.2.5, the number of cartons equal to the number of bottles in a set (*see* 5.3.3) shall be taken at random from these cartons and then the required number of bottles picked up according to 5.3.1.

5.3.3 The sample bottles selected as in 5.3.1 or 5.3.2 shall be divided at random into three equal sets and labelled with all the particulars of sampling. One of these sets of sample bottles shall be for the purchaser, another for the vendor and the third for referee. The sample bottles to be tested for net volume shall be kept separately.

5.3.4 Referee Sample — Referee sample shall consist of a set of sample bottles marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier so as to be used in case of a dispute between the two.

5.4 Number of Tests

5.4.1 Beer and Wines — Tests for carbon dioxide, microbiological requirements pH and organoleptic requirements in this sequence shall be carried out on the individual sample bottles selected according to col 2 of Table 1. The net volume shall be tested on each of the individual bottles selected in each set (*see* 5.3.3). Tests for the remaining requirements of this specification shall be conducted on the composite sample prepared by thoroughly mixing approximately equal quantity of material from the individual sample bottles selected a in each set (*see* 5.3.3).

5.4.2 Whisky, Gin, Brandy, Rum — Tests for ethyl alcohol content by hydrometer method shall be carried out on individual sample bottles selected in each set (*see* 5.3.3) and net volume shall be tested on each of the individual bottles selected in each set (*see* 5.3.3). Organoleptic requirements shall also be tested on individual sample bottles. Tests for remaining requirements of the relevant specification shall be carried out on the composite sample prepared by thoroughly mixing approximately equal quantity of material from the individual sample bottles selected in each set (*see* 5.3.3).

5.5 Criteria for Conformity — The lot shall be declared as conforming to the requirements of the relevant specification if 5.5.1, 5.5.2, 5.5.3 and 5.5.4 are satisfied.

5.5.1 For those characteristics tested on the composite sample, all the test results shall satisfy the corresponding specification requirements.

5.5.2 For each characteristic other than organoleptic and microbiological

tested on the individual samples, the average (\bar{X}) and range (R) shall be computed as follows :

$$\text{Average } (\bar{X}) = \frac{\text{The sum of test results}}{\text{Number of test results}}$$

$$\text{Range } (R) = \text{Difference between the maximum and the minimum values of the test results}$$

5.5.2.1 If the specification limit for the characteristic is given as minimum, the value of expression ($\bar{X} - 0.6R$) shall be calculated from the relevant test results. If the value so obtained is greater than or equal to the minimum limit, the lot shall be deemed to have conformed to the requirements of that characteristic.

5.5.2.2 If the specification limit for the characteristic is given as a maximum, the value of expression ($\bar{X} + 0.6R$) shall be calculated from the relevant test results. If the value so obtained is less than or equal to the maximum limit, the lot shall be considered as satisfying the requirements of that characteristic.

5.5.2.3 If the characteristic has two sided specification limits, the values of the expression ($\bar{X} - 0.6R$) and ($\bar{X} + 0.6R$) shall be calculated from the relevant test results. If the values so obtained lie between the two specification limits, the lot shall be considered as meeting the requirements of that characteristic.

5.5.3 For organoleptic and microbiological requirements, if all the test results on each of the individual samples satisfy the relevant specification requirements, the lot shall be considered as satisfying the requirements of that characteristic.

5.5.4 For net volume the bottles selected from each set (*see 5.4*) meant for testing net volume shall be randomly divided into groups of 12 bottles each. The net volume shall be the volume indicated on the bottle subject to the tolerance for individual bottle given in the relevant specification. The total volume of 12 bottles shall also be examined according to relevant tolerances. The lot shall be deemed to have satisfied the requirement of net volume if **5.5.4.1** and **5.5.4.2** are satisfied.

5.5.4.1 Each individual sample bottle shall satisfy the requirement of net volume indicated on the bottle subject to the corresponding tolerances given in the individual material specification.

5.5.4.2 Each group of 12 bottles shall satisfy the requirement of net volume subject to the corresponding tolerances given in the individual material specification.

APPENDIX A

(Clause 4.3.1.1)

RECOMMENDED FREQUENCY OF TESTING AT DIFFERENT STAGES OF MANUFACTURE OF BEER

SL No.	STAGE	CHARACTERISTICS	FREQUENCY
i)	Wort boiling	Specific gravity	One sample from each wort kettle
ii)	Meshing	Specific gravity	One sample from each mesh tank
iii)	Fermentation	Specific gravity and temperature	One sample from each fermentor
iv)	Bottling tank	Colour, clarity, ethyl alcohol content, pH and carbon dioxide	A composite sample from each batch after the material is filtered and carbonized. For this purpose, small portions of material drawn periodically at every two hours shall be mixed thoroughly
v)	Storage	Specific gravity, colour and pH	One sample from each storage tank at regular intervals
vi)	Bottling machine	a) Organoleptic and ethyl alcohol content	a) Three samples at regular intervals per shift from each machine
		b) Microbiological requirements	b) One sample per shift from each machine
		c) Net volume	c) 12 bottles per hour in each shift
vii)	Pasteurization	Temperature control for effective pasteurization	Three samples per shift from each machine

APPENDIX B

(Clause 4.3.2.1)

RECOMMENDED FREQUENCY OF TESTING AT DIFFERENT STAGES OF MANUFACTURE OF WHISKY, RUM, GIN AND BRANDY

SL No.	STAGE	CHARACTERISTICS	FREQUENCY
i)	Meshing	Dilution and specific gravity of molasses solution	One sample from each mesh tank
ii)	Fermentation	Temperature and specific gravity	One sample from each fermentation tank
iii)	Distillation	All requirements given in the relevant product specification except maturation and net volume	A composite sample prepared by mixing portions of material taken at different intervals from last distillation column in a shift
iv)	Maturation	Quality of maturation	One sample every three months from each wooden cask or barrel or vat
v)	Bottling	a) Colour, clarity and ethyl alcohol content (by pycnometric or hydrometer method as per the requirements)	a) One representative sample from each batch
		b) Net volume	b) One sample every half-an-hour

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	Unit	Symbol	Definition
Force	newton	N	1 N = 1 kg.m/s ²
Energy	Joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s ⁻¹)
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²

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